

# MEMORANDUM

TO: Mr. Addison Rice  
Anderson, Mulholland and Associates

DATE: July 27, 2016

FROM: R. Infante 

FILE: 1607228-1607235

RE: Data Validation  
Air samples

SDG: 1607235A; 1607235B; 1607235C; ~~1606298D~~; 1607228A 

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## SUMMARY

Full validation was performed on the data for one (1) gas sample analyzed for volatile organic compounds (full suite) and methanol by method Compendium Method TO-15: Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999. Methane analyzed by ASTM method D-1946-modified. Naphthalene by method Compendium Method TO-17: Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes, January 1999. The samples were collected at the Bristol Myer Squib, Humacao, PR site on July 9, 2016 and submitted to Eurofins Air Toxics, Inc. of Folsom, California that analyzed and reported the results under delivery groups (SDG) 1607235A; 1607235B; 1607235C; ~~1606298D~~; and 1607228A.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Compendium Method TO-15. Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999; Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006. The QC criteria of methods TO-17 and ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use. Results for tetrahydrofuran was qualified as estimated (UJ) in sample 1607235A due to continuing calibration check % difference outside method performance limit.

## SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
=====				
B730IA-1 (070816)	1607235A-07A	07/09/16	Air	TO-15 (full suite)
B730IA-1 (070816)	1607235B-07A	07/09/16	Air	TO-15 (methanol)
B730IA-1	1607228A-07A	07/09/16	Air	TO-17 (naphthalene)
B730IA-1 (070816)	1607235C-07A	07/09/16	Air	ASTM D-1946 (methane)

## REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Gas chromatography/mass spectrometry (GC/MS) tunes
- o Initial and continuing calibrations
- o Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria
- o Surrogate spike recovery
- o Internal standard performance and retention times
- o Field duplicate results
- o Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- o Quantitation limits and sample results

## DISCUSSION

### Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

The Chain of Custody (COC) information for sample B7IA-1D(061016) did not match the entry on the sample tag with regard to sample identification. The information on the COC was used to process and report the sample.

### Holding Times and Sample Preservation

Sample preservation was acceptable. Samples received in good conditions.

Samples analyzed within method recommended holding time.

## GC/MS Tunes

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

## Initial and Continuing Calibrations

### VOCs – (Method TO-15-full suite)

Initial calibration meets method performance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard, continuing calibration meet the method performance criteria except for the following analytes:

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibration met the method performance criteria except the cases described in this document.				
MSD-17				
07/14/16	1607235A-09A	-38 %	Tetrahydrofuran	1607235A-07A

Results qualified estimated (J) or (UJ) in affected samples.

### VOCs – (Method TO-15-methanol)

A one point initial calibration meets method performance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard, continuing calibration meet the method performance criteria.

### VOCs – (Method TO-17-naphthalene)

Initial calibration meets method performance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard, continuing calibration meet the method performance criteria.

### VOCs – (Method ASTM D-1946-modified - methane)

Initial calibration meets method performance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard, continuing calibration meet the method performance criteria

## Method Blank/Trip Blank/Field Blank

Several VOCs TO-15 (full suite) analytes were detected in the method blanks analyzed below the reporting limit/action level. Laboratory qualified the results as estimated (J) in the method blanks. No further qualification made.

No sample analytes were detected in methods blanks analyzed for naphthalene, methanol and methane.

Summa canister met cleaning certification criteria.

No trip/field blank analyzed with this data package.

### **Surrogate Spike Recovery**

The surrogate recoveries as per method TO-15, TO-17 and ASTM D-1946 were within the laboratory QC acceptance limits in all samples analyzed.

### **Internal Standard Performance**

#### **VOCs – TO-15 and TO-17**

Samples were spiked with the method specified internal standard. Internal standard are performance and retention times met the QC acceptance criteria in all sample analyses and calibration standards.

### **Laboratory/Field Duplicate Results**

Laboratory duplicates (LCS/LCSD) were analyzed as part of this data set. Target analytes meet the RPD performance criteria of + 25 % for analytes 5 x SQL .

### **LCS/LCSD Results**

LCS/LCSD (blank spike) analyzed by the laboratory associated with this data package; % recoveries and RPD within laboratory and generally acceptable control limits.

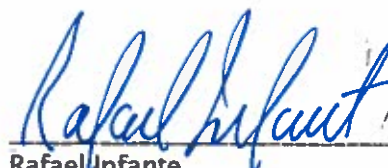
### **Quantitation Limits and Sample Results**


Dilutions were not performed on TO-15 samples (see worksheet).

Calculations were spot checked.

### **Certification**

The samples reported on SDG: 1607235A; 1607235B; 1607235C; and 1607228A and described in the sample table were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid some of the results were qualified.

  
Rafael Infante  
Chemist License 1888





## Air Toxics

Client Sample ID: B301A-1 (070816)

Lab ID#: 1607235A-07A

### MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name:	e071418	Date of Collection:	7/9/16 6:12:00 PM	
Dil. Factor:	1.64	Date of Analysis:	7/14/16 09:54 PM	
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Freon 12	0.16	0.39	0.81	1.9
Freon 114	0.16	Not Detected	1.1	Not Detected
Chloromethane	0.82	0.60 J	1.7	1.2 J
Vinyl Chloride	0.16	Not Detected	0.42	Not Detected
1,3-Butadiene	0.16	Not Detected	0.36	Not Detected
Bromomethane	0.82	Not Detected	3.2	Not Detected
Chloroethane	0.82	Not Detected	2.2	Not Detected
Freon 11	0.16	0.26	0.92	1.4
Ethanol	0.82	5.8	1.5	11
Freon 113	0.16	0.070 J	1.2	0.54 J
1,1-Dichloroethene	0.16	Not Detected	0.65	Not Detected
Acetone	0.82	6.8	1.9	16
2-Propanol	0.82	3.0	2.0	7.3
Carbon Disulfide	0.82	0.65 J	2.6	2.0 J
3-Chloropropene	0.82	Not Detected	2.6	Not Detected
Methylene Chloride	0.33	0.074 J	1.1	0.26 J
Methyl tert-butyl ether	0.16	Not Detected	0.59	Not Detected
trans-1,2-Dichloroethene	0.16	Not Detected	0.65	Not Detected
Hexane	0.16	Not Detected	0.58	Not Detected
1,1-Dichloroethane	0.16	Not Detected	0.66	Not Detected
2-Butanone (Methyl Ethyl Ketone)	0.82	1.2	2.4	3.6
cis-1,2-Dichloroethene	0.16	Not Detected	0.65	Not Detected
Tetrahydrofuran	0.82	Not Detected UJ	2.4	Not Detected UJ
Chloroform	0.16	Not Detected	0.80	Not Detected
1,1,1-Trichloroethane	0.16	Not Detected	0.89	Not Detected
Cyclohexane	0.16	Not Detected	0.56	Not Detected
Carbon Tetrachloride	0.16	0.066 J	1.0	0.42 J
2,2,4-Trimethylpentane	0.82	Not Detected	3.8	Not Detected
Benzene	0.16	0.073 J	0.52	0.23 J
1,2-Dichloroethane	0.16	Not Detected	0.66	Not Detected
Heptane	0.16	Not Detected	0.67	Not Detected
Trichloroethene	0.16	Not Detected	0.88	Not Detected
1,2-Dichloropropane	0.16	Not Detected	0.76	Not Detected
1,4-Dioxane	0.16	Not Detected	0.59	Not Detected
Bromodichloromethane	0.16	Not Detected	1.1	Not Detected
cis-1,3-Dichloropropene	0.16	Not Detected	0.74	Not Detected
4-Methyl-2-pentanone	0.16	0.12 J	0.67	0.49 J
Toluene	0.16	0.18	0.62	Not Detected
trans-1,3-Dichloropropene	0.16	Not Detected	0.74	Not Detected
1,1,2-Trichloroethane	0.16	Not Detected	0.89	Not Detected
Tetrachloroethene	0.16	Not Detected	1.1	Not Detected
2-Hexanone	0.82	0.17 J	3.4	0.70 J



## Air Toxics

Client Sample ID: B301A-1 (070816)

Lab ID#: 1607235A-07A

### MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name:	e071418	Date of Collection: 7/9/16 6:12:00 PM		
Dil. Factor:	1.64	Date of Analysis: 7/14/16 09:54 PM		
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Dibromochloromethane	0.16	Not Detected	1.4	Not Detected
1,2-Dibromoethane (EDB)	0.16	Not Detected	1.3	Not Detected
Chlorobenzene	0.16	Not Detected	0.76	Not Detected
Ethyl Benzene	0.16	Not Detected	0.71	Not Detected
m,p-Xylene	0.16	0.12 J	0.71	0.54 J
o-Xylene	0.16	Not Detected	0.71	Not Detected
Styrene	0.16	Not Detected	0.70	Not Detected
Bromoform	0.16	Not Detected	1.7	Not Detected
Cumene	0.16	Not Detected	0.81	Not Detected
1,1,2,2-Tetrachloroethane	0.16	Not Detected	1.1	Not Detected
Propylbenzene	0.16	Not Detected	0.81	Not Detected
4-Ethyltoluene	0.16	Not Detected	0.81	Not Detected
1,3,5-Trimethylbenzene	0.16	Not Detected	0.81	Not Detected
1,2,4-Trimethylbenzene	0.16	Not Detected	0.81	Not Detected
1,3-Dichlorobenzene	0.16	Not Detected	0.99	Not Detected
1,4-Dichlorobenzene	0.16	Not Detected	0.99	Not Detected
alpha-Chlorotoluene	0.16	Not Detected	0.85	Not Detected
1,2-Dichlorobenzene	0.16	Not Detected	0.99	Not Detected
1,2,4-Trichlorobenzene	0.82	Not Detected	6.1	Not Detected
Hexachlorobutadiene	0.82	Not Detected	8.7	Not Detected
Naphthalene	0.82	Not Detected	4.3	Not Detected

J = Estimated value.

UJ = Analyte associated with low bias in the CCV.

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	108	70-130
Toluene-d8	97	70-130
4-Bromofluorobenzene	98	70-130





## Air Toxics

Client Sample ID: B301A-1 (070816)

Lab ID#: 1607235B-07A

### EPA METHOD TO-15 GC/MS

File Name:	14071920	Date of Collection:	7/9/16 6:12:00 PM
Dil. Factor:	1.64	Date of Analysis:	7/19/16 04:13 PM

Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)
Methanol	160	Not Detected	210	Not Detected

Container Type: 6 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Method Limits
1,2-Dichloroethane-d4	103	70-130
Toluene-d8	102	70-130
4-Bromofluorobenzene	96	70-130





## Air Toxics

Client Sample ID: B301A-1 (070816)

Lab ID#: 1607235C-07A

### NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	10071421	Date of Collection:	7/9/16 6:12:00 PM
Dil. Factor:	1.64	Date of Analysis:	7/14/16 07:36 PM
Compound	Rpt. Limit (%)	Amount (%)	
Methane	0.00016	0.00037	

Container Type: 6 Liter Summa Canister (100% Certified)





## Air Toxics

Client Sample ID: B30IA-1

Lab ID#: 1607228A-07A

### EPA METHOD TO-17

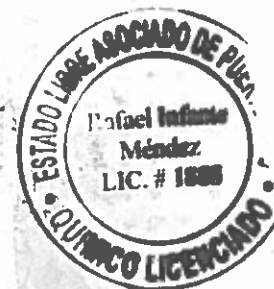
File Name:	6071409	Date of Extraction: NA	Date of Collection: 7/9/16 6:10:00 PM
Dil. Factor:	1.00		Date of Analysis: 7/14/16 07:29 PM

Compound	Rpt. Limit (ppbv)	Rpt. Limit (ug/m3)	Amount (ppbv)	Amount (ug/m3)
Naphthalene	0.0090	0.047	0.017	0.087

Air Sample Volume(L): 21.1

Container Type: TO-17 VI Tube

Surrogates	%Recovery	Method Limits
Naphthalene-d8	112	50-150



## TO-17 SAMPLE COLLECTION

# Air Toxics LTD.

## CHAIN-OF-CUSTODY RECORD

## Sample Transportation Notice

Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4822.

180 BLUE RAVINE ROAD, SUITE B  
FOLSOM, CA 95630

(916) 985-1000 FAX (916) 985-1020

Page 1 of 2

Project Manager Terry Taylor

Collected by: (Print and Sign) RC, TT, DL

Company AMA Email ttaylor@amiconsult.com

Address 720 Westchester Ave City Purchase State NY Zip 10577

Phone 914-251-0400 x309 Fax 914-251-1286

## Project Info:

P.O. # \_\_\_\_\_

Project # B45 B18 V1

Project Name \_\_\_\_\_

## Turn Around Time:

☒ Normal ☐ Rush

B30IA-1

specify

## Reporting Units:

☐ ppmv

☒ ppbv

☒ ug/m3

☐ mg/m3

Lab ID	Field Sample I.D. (Location)	Engraved or Stamped Tube #	Date of Collection (mm/dd/yy)	Start Time (hr:min)	End Time (hr:min)	Pre-Test Flow Rate	Post-Test Flow Rate	Volume ml	Indoor/Outdoor		Indoor Air	Outdoor Air	Soil Vapor	Other
									% RH	Temp				
	B18IA-1	151814	7/9/16	1803	0952	35	35	11,100	85	80	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18IA-2	151188	7/9/16	1852	1852	36	34	16,800	86	80	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18IA-3	150090	7/9/16	1827	1827	36	38	17,700	98	79	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18IA-4	153639	7/9/16	1836	1836	34	33	16,080	88	78	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18IA-5	150877	7/9/16	1844	1844	35	34	16,560	79	83	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18IA-1D	149733	7/9/16	1803	1051	35	33	11,420	85	60	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	*B30IA-1*	137170	7/9/16	1810	1810	36	52	23,120	81	81	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18AA-070816	149726	7/11/16	1145	1145	34	36	16,800	75	87	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	B18SS-1	149703	7/11/16	1746	1749	134	134	402	77	75	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
	B18SS-2	149853	7/12/16	1938	1941	133	134	402	70	74	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

Relinquished by: (signature)

Date/Time

07-13-16 1700

Received by: (signature)

Date/Time

Fed Ex

Relinquished by: (signature)

Date/Time

Received by: (signature)

Date/Time

7/14/16

Relinquished by: (signature)

Date/Time

Received by: (signature)

Date/Time

1030

Notes:

\* 24 hour turnaround time for B30IA-1. All other samples are normal turnaround. Report only NAF.

Lab Use Only	Shipper Name	Air Bill #	Temp (C)	Condition	Custody Seal Intact?	Work Order #
	<u>Fed Ex</u>		<u>9.8°C</u>	<u>SDR</u>	Yes No <u>None</u>	<u>1607228</u>

# DATA REVIEW WORKSHEETS

Project Number: 1607235A  
Date: 07/09/2016

## REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from "Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999"; USEPA Hazardous Waste Support Branch. Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins - Air Toxics data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1607235A Sample matrix: Air  
No. of Samples: 1

Trip blank No.: -  
Field blank No.: -  
Equipment blank No.: -  
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input checked="" type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input type="checkbox"/> N/A Matrix Spike/Matrix Spike Duplicate	

Overall Comments: VOCs\_by\_method\_TO-15\_(full suite)

### Definition of Qualifiers:

J- Estimated results  
U- Compound not detected  
R- Rejected data  
UJ- Estimated nondetect

Reviewer: Rafael Lafont  
Date: 07/27/2016

M

D.

D

This image shows a single sheet of white paper with horizontal blue or grey ruling lines. A dashed diagonal line runs across the page from the upper left to the lower right, likely serving as a guide for folding. The paper appears to be part of a notebook or a template for a project.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. Samples received in good conditions.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples ( $\text{pH} \leq 2$ ,  $4^{\circ}\text{C}$ ), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples,  $4^{\circ}\text{C}$ , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria:  $4 \pm 2^{\circ}\text{C}$ ): N/A – summa canisters

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is  $< 10\%$ , estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but  $< 14$  days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but  $< 28$  days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded ( $> 28$  days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ( $> 10^{\circ}\text{C}$ ), estimate positive results (J) and nondetects (UJ).

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met see below \_\_\_\_\_

## GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

  X   The BFB performance results were reviewed and found to be within the specified criteria.

  X   BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:

**If mass calibration is in error, all associated data are rejected.**

**If mass calibration is in error, all associated data are rejected.**

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below X

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 03/17/16  
 Dates of continuing calibration: 07/14/16  
 Instrument ID numbers: MSD-E  
 Matrix/Level: Air/low

DATE	LAB FILE ID#	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibration met the method performance criteria except for the following:				
07/14/16	1607235A-09A	-38 %	Tetrahydrofuran	1607235A-07A

**Note:** Samples results qualified as estimated (J) in affected sample.

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.

All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.

All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a % D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a % D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).

If any compound has a % D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has r  $< 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

# DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below   X  

## V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION/UNITS
None of the analyte detected in the method/laboratory blank above the reporting limit/action level for blanks.				

**Note:** Several analytes detected in the method blanks analyzed on 07/14/16 below the reporting limit/action level. Laboratory qualified the results as estimated (J). No further qualification made.

Summa canisters met cleaning certification criteria

### Field/Equipment/Trip blank

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
No field/trip/equipment blanks analyzed with this data package.				

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below X

### V B. BLANK ANALYSIS RESULTS (Section 3)

## Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

**Specific actions are as follows:**

If the concentration is < sample quantitation limit (SQL) and  $\leq$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

**Notes:**

### High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND			ACTION
	1,2-DICHLOROETHANE- d4	Toluene- d8	4-BFB	

  Surrogate recoveries within laboratory control limits    
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

QC Limits\* (Air)

      LL to UL      70 to 130                  70 to 130      70 to 130      

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

# DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below N/A

## VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level: \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
_____ MS/MSD are not required as part of Method TO-15; blank spike used to assess _____ accuracy _____					

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below N/A

## VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

### MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

This image shows a single sheet of white paper with horizontal blue or grey ruling lines. A dashed diagonal line runs across the page from the bottom-left corner towards the top-right corner. The paper appears to be part of a notebook or a template for writing.

**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
_LCS/LCSD_%_recoveries_and_RPD_within_laboratory_control_limits._			

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (J) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### IX. LABORATORY DUPLICATE PRECISION

Sample IDs:   LCS/LCSD\_(07/14/2016)  

Matrix:   Air  

Laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD  $\pm$  25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD within laboratory and generally acceptable control limits.					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
------	-----------	--------	---------	------------------	--------

Internal standard area and retention times within laboratory control limits for both samples and calibration standards


Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1607235A-07A

2-Propanol                      RF = 1.69275

$$[ ] = (67546)(5.0)/(110110)(1.69275)$$

$$= 1.812 \text{ ppbv OK}$$

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### XII. QUANTITATION LIMITS

#### A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples were diluted by a factor of 1.64 x.		

#### B. Percent Solids

List samples which have  $\leq 50$  % solids

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#### Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)

# DATA REVIEW WORKSHEETS

Project Number: 1607235B  
Date: 07/09/2016

## REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from "Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999"; USEPA Hazardous Waste Support Branch. Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins - Air Toxics data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1607235B Sample matrix: Air  
No. of Samples: 1

Trip blank No.: -  
Field blank No.: -  
Equipment blank No.: -  
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input checked="" type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input type="checkbox"/> N/A Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Methanol by method TO-15

### Definition of Qualifiers:

J- Estimated results  
U- Compound not detected  
R- Rejected data  
UJ- Estimated nondetect

Reviewer: Rafael Defaut  
Date: 07/27/2016

## DATA REVIEW WORKSHEETS

## DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED \_\_\_\_\_

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met \_\_\_\_\_  
and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH  $\leq$  2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4  $\pm$  2 °C): N/A – summa canisters

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met see below \_\_\_\_\_

## GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

X The BFB performance results were reviewed and found to be within the specified criteria.

  X   BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

**List the samples affected:**

**If mass calibration is in error, all associated data are rejected.**

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 07/19/2016  
 Dates of continuing calibration: 07/19/2016  
 Instrument ID numbers: MSD-14  
 Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
One point calibration. Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.					

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.  
 All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.  
 All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.  
 Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.  
 If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.  
 If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has a % D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has a % D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).  
 If any compound has a % D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has  $r < 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below \_\_\_\_\_

### V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

#### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

#### Field/Equipment/Trip blank

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

## DATA REVIEW WORKSHEETS

All criteria were met ☒  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### V B. BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and  $\leq$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES

# DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

## SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND			ACTION
	1,2-DICHLOROETHANE- d4	Toluene- d8	4-BFB	

  Surrogate recoveries within laboratory control limits    
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

QC Limits\* (Air)

      LL  to  UL      70  to  130                                  70  to  130      70  to  130      

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below \_\_N/A\_\_

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level: \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
____MS/MSD are not required as part of Method TO-15; blank spike used to assess accuracy____					

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below N/A

#### VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

### MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

[illegible]

**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
___No LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package._____			

\* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

\* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below \_\_\_\_\_ N/A \_\_\_\_\_

### IX. LABORATORY DUPLICATE PRECISION

Sample IDs: \_\_\_\_\_ - \_\_\_\_\_

Matrix: \_\_\_\_\_ - \_\_\_\_\_

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD  $\pm$  25% for air samples. If both samples and duplicate are  $<5$  SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No laboratory/field duplicate analyzed with this data package					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
------	-----------	--------	---------	------------------	--------

Internal standard area and retention times within laboratory control limits for both samples and calibration standards


#### Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

Calibration check

1,2-Dichloroethane-d4              RF = 2.08040

$$[ ] = (255976)(400)/(119088)(2.08040)$$

$$= 413.3 \text{ ppbv OK}$$

## XII. QUANTITATION LIMITS

**A. Dilution performed**

### B. Percent Solids

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

13b

## DATA REVIEW WORKSHEETS

Project Number: 1607228A

Date: 07/09/2016

### REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from "Compendium Method TO-15. Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999"; USEPA Hazardous Waste Support Branch. Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins - Air Toxics data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1607228A

Sample matrix: Air

No. of Samples: 1

Trip blank No.: -

Field blank No.: -

Equipment blank No.: -

Field duplicate No.: -

☒ Data Completeness

☒ Holding Times

☒ GC/MS Tuning

☒ Internal Standard Performance

☒ Blanks

☒ Surrogate Recoveries

☐ N/A Matrix Spike/Matrix Spike Duplicate

☒ Laboratory Control Spikes

☒ Field Duplicates

☒ Calibrations

☒ Compound Identifications

☒ Compound Quantitation

☒ Quantitation Limits

Overall Comments: Naphthalene\_by\_method\_TO-17

#### Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated nondetect

Reviewer: Rafael Infante

Date: 07/27/2016

## DATA REVIEW WORKSHEETS

## DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. Temperature was measured and was not within $4 \pm 2$ °C. Coolant in the form of blue ice was present. Analysis proceeded. No action taken, samples analyzed within 7 days of collection.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples ( $\text{pH} \leq 2$ ,  $4^{\circ}\text{C}$ ), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples,  $4^{\circ}\text{C}$ , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria:  $4 \pm 2$  °C):  $16.2^{\circ}\text{C}$

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is  $< 10\%$ , estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but  $< 14$  days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but  $< 28$  days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded ( $> 28$  days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ( $> 10^{\circ}\text{C}$ ), estimate positive results (J) and nondetects (UJ).

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met see below \_\_\_\_\_

## GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

  X   The BFB performance results were reviewed and found to be within the specified criteria.

  X   BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:

**If mass calibration is in error, all associated data are rejected.**

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 07/12-13/16

Dates of continuing calibration: 07/14/16

Instrument ID numbers: MSD-6

Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements. Desorption efficiency verification for Naphthalene was 99.8 %; meet method specific requirements.					

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.

All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.

All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a % D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a % D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).

If any compound has a % D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has r  $< 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

**V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)**

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

**List the contamination in the blanks below. High and low levels blanks must be treated separately.**

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

**Field/Equipment/Trip blank**

[illegible]

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

## Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

**ALs = 5x for any other compounds**

**Specific actions are as follows:**

If the concentration is < sample quantitation limit (SQL) and  $\leq$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

**Notes:**

**High and low level blanks must be treated separately**

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met \_\_\_\_\_  
 and/or see below \_\_\_\_\_

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND			ACTION
	1,2-DICHLOROETHANE- d4	Toluene- d8	4-BFB	

Surrogate recoveries within laboratory control limits \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

QC Limits\* (Air)

\_\_\_\_\_ LL to UL \_\_\_\_\_ to \_\_\_\_\_      \_50\_ to \_150\_ \_\_\_\_\_ to \_\_\_\_\_

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below N/A

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level: \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
MS/MSD are not required as part of Method TO-17; blank spike used to assess accuracy					

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below N/A

## VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

### MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

**List the %RSD of the compounds which do not meet the criteria.**

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
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This image shows a full page of blank handwriting practice paper. It features multiple sets of horizontal lines. Each set consists of a solid top line, a dashed middle line, and a solid bottom line, providing a guide for letter height and placement. The lines are evenly spaced across the entire page.

**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

# DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met \_\_\_\_\_  
and/or see below \_\_\_\_\_

## VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
____LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package;_%_recoveries_and_RPD_____			
____within_laboratory_control_limits._____			

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

All criteria were met X  
Criteria were not met  
and/or see below

Sample IDs: \_LCS/LCSD\_(laboratory) Matrix: Air

**The project QAPP should be reviewed for project-specific information.**

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory duplicate analyzed with data package. LCS/LCSD % recoveries RPD used to assess precision. RPD within the method performance criteria.					

**If both sample and duplicate results are not detected, no action is needed.**

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
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Internal standard area and retention times within laboratory control limits for both samples and calibration standards


Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1607228A-07A

Naphthalene

RF = 1.93557

$$[ ] = (43615)(36)/(440102)(1.93557)$$

$$= 1.843 \text{ ng OK}$$

## DATA REVIEW WORKSHEETS

All criteria were met ☒ X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### XII. QUANTITATION LIMITS

#### A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
No dilution performed.		

#### B. Percent Solids

List samples which have  $\leq 50\%$  solids

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#### Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is  $< 10\%$ , estimate positive results (J) and reject nondetects (R)

# DATA REVIEW WORKSHEETS

Project Number: 1607235C

Date: 07/09/2016

## REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from ASTM D-1946 method for measuring permanent gases and light hydrocarbons in refinery and other sources samples using gas chromatography (GC) and a thermal conductivity detector (TCD) and/or flame ionization detection (FID). Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1607235C

Sample matrix: Air

No. of Samples: 1

Trip blank No.: -

Field blank No.: -

Equipment blank No.: -

Field duplicate No.: -

☒ Data Completeness

☒ Laboratory Control Spikes

☒ Holding Times

☒ Field Duplicates

☐ N/A GC/MS Tuning

☒ Calibrations

☐ N/A Internal Standard Performance

☒ Compound Identifications

☒ Blanks

☒ Compound Quantitation

☐ N/A Surrogate Recoveries

☒ Quantitation Limits

☐ N/A Matrix Spike/Matrix Spike Duplicate

Overall Comments: Methane\_by ASTM\_method\_D-1946\_(modified)

### Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated nondetect

Reviewer: Rafael Lafont

Date: 07/27/2016

## DATA REVIEW WORKSHEETS

## DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples ( $\text{pH} \leq 2$ ,  $4^{\circ}\text{C}$ ), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples,  $4^{\circ}\text{C}$ , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria:  $4 \pm 2^{\circ}\text{C}$ ): N/A – summa canisters

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ).

If the % solid of soil samples is  $< 10\%$ , estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but  $< 14$  days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but  $< 28$  days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded ( $> 28$  days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ( $> 10^{\circ}\text{C}$ ), estimate positive results (J) and nondetects (UJ).

## DATA REVIEW WORKSHEETS

All criteria were met NA  
Criteria were not met see below \_\_\_\_\_

### GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

NA The BFB performance results were reviewed and found to be within the specified criteria.

NA BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List \_\_\_\_\_ the \_\_\_\_\_ samples \_\_\_\_\_ affected:

If mass calibration is in error, all associated data are rejected.

Note: Samples analyzed using GC with either TCD or FID detection.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 01/15/16

Dates of continuing calibration: 07/14/16

Instrument ID numbers: GC-10

Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.					

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.

All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.

All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a % D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has a % D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).

If any compound has a % D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).

If any compound has r  $< 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

**V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)**

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

**Field/Equipment/Trip blank**

[illegible]

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

## Blank Actions

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

**Specific actions are as follows:**

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

**Notes:**

### High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met   N/A    
 Criteria were not met  
 and/or see below           

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
-----------	--------------------	--------

  Surrogate standards not required by the method.  

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

QC Limits\* (Air)

\_\_\_\_\_ LL to UL \_\_\_\_\_ to \_\_\_\_\_ to \_\_\_\_\_ to \_\_\_\_\_

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below N/A

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ - \_\_\_\_\_ Matrix/Level: \_\_\_\_\_ - \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
<u>MS/MSD are not required as part of ASTM-method D-1946; blank spike used to assess accuracy</u>					

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (JJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below \_\_\_\_\_ N/A \_\_\_\_\_

## VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

### MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
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[illegible]

**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
____LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package;_recoveries_and_RPD____			
____within_laboratory_control_limits.____			

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met ☒  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample ID \_\_\_\_\_ LCS/LCSD\_(07/14/16)\_\_\_\_\_

Matrix: Air

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD  $\pm$  25% for air samples. If both samples and duplicate are  $<5$  SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
No field/laboratory duplicates analyzed as part of this data package. LCS/LCSD % recoveries RPD used to assess precision. RPD LCS/LCSD within laboratory control limits.					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

All criteria were met   N/A    
 Criteria were not met  
 and/or see below           

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
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Internal standard not required by the method. Samples quantified by external standard method


Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met ☒   
Criteria were not met   
and/or see below \_\_\_\_\_

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

Blank Spike (LCS)

Methane RF = 226379851

$$[ ] = (2334071784)/(226379851)$$

= 10.3 % OK

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### XII. QUANTITATION LIMITS

#### A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples diluted by a factor of less than 1.64		

#### B. Percent Solids

List samples which have  $\leq 50$  % solids

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#### Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)